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Cafestol extraction yield from different coffee brew mechanisms

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ABSTRACT

The extraction yield of cafestol from roast and ground (R&G) coffee beans was evaluated using brews prepared by four brewing mechanisms (boiled, Turkish, French Press and Mocha Pot). The cafestol content of the R&G coffee and the resulting brews was measured and extraction yield calculated. The R&G coffee had an average cafestol content of 603 mg/100 g R&G coffee with a slight reduction at higher roast intensities. In the brews, preparation method had an impact on cafestol concentration with French, Turkish and boiled preparation methods producing the highest cafestol concentrations. The extraction yield of cafestol was shown to be dependent on the brew mechanism and roasting time, with the lightest roast coffee prepared by French press or boiled preparations having the highest cafestol extraction yield (6.5% and 5.84%) and dark roast Mocha and Turkish preparations had the lowest extraction yields of 2.42% and 2.88% respectively.

1. Introduction

Coffee is a globally consumed beverage and is prepared in a wide variety of formats including Scandinavian-type boiled coffee, drip filtered coffee, instant or soluble coffee and espresso. Within each class of brew preparation method, individual population groups consume coffee in a range of formats (e.g. 37 °C–88 °C Lee, Carstens, & O'Mahony, 2003), 0%–80% milk (Lee & O'Mahony, 2002), 0 g–16 g of sugar, 25 mL–880 mL in volume (Hsu & Hung, 2005), with or without milk, foamed milk, cream, ice, flavourings, brew adjuncts or co-adjuncts (Fisk, Massey, & Hansen, 2011; Massey, Fisk, & Henson, 2011).

Coffee brew contains a wide range of components including medium- to long-chain polysaccharides, melanoidins, volatile aroma compounds and lipid like compounds with a range of positive, negative and neutral health benefits (Esquivel & Jiménez, 2012). Coffee also contains a number of diterpenes including cafestol, which has been shown to have cholesterol raising properties (Butt & Sultan, 2011) and is proposed to increase serum cholesterol by 1 mg/dL for each 2 mg of consumed cafestol, although this has not necessarily been proven in all population groups (Weusten-van der Wouw et al., 1994).

The varied format and highly variable size and frequency of consumption make prediction of risk factors, such as hypertension from caffeine consumption and elevated cholesterol levels from the consumption of diterpenes, challenging for health authorities and manufacturers.

The cafestol content of a standard cup of coffee varies depending on brew mechanism but is highest in unfiltered preparation methods such as Scandinavian-type boiled coffee and Turkish coffee with up to 88.7 mg/L in some Turkish brews (Table 1) (Gross, Jaccaud, & Huggett, 1997; Urgert et al., 1995). Filtered coffees such as drip-filter and soluble coffee contain negligible levels of cafestol in the brew, as the paper filter in drip filtered coffee retains the diterpenes and in soluble coffee the diterpenes are retained with the grounds during production (Gross et al., 1997).

Values for cafestol concentration by brew mechanism from previous studies (Table 1) are often variable due to differing extraction parameters (Eulitz, Kolling-Speer, & Speer, 1999), grind sizes (Buchmann, Zahm, Kolling-Speer, & Speer, 2010; Kurzrock & Speer, 2001; Sehat, Montag, & Speer, 1993), coffee to water ratios (Buchmann et al., 2010), temperatures (Buchmann et al., 2010) and brewing technologies e.g. coffee pads (Boekschoten, Van Cruchten, Kosmeijer-Schuil, & Katan, 2006).

Cafestol is not extracted by a simple dissolution kinetics, when hot water interacts with R&G coffee a number of phenomena occur (Lee, Kempthorne, & Hardy, 1992; Merritt & Proctor, 1958), firstly the highly soluble components dissolve in the water phase and are extracted, for example organic acids (Lentner & Deatherage, 1958), secondly less soluble or physically entrapped compounds (e.g. arabinogalactan) (Redgwell & Fischer, 2006) are forced out by physical mechanisms, thirdly the heat leads to thermal degradation making select components more soluble and therefore more available for extraction (e.g. galactomannan) and fourthly mobile water will physically lift and migrate coffee fines and emulsify coffee oil into suspension (Escher, Schenker, Handschin, Frey, & Perren, 2000; Eulitz et al., 1999); it is these components (coffee fines and coffee oil) that contain the cafestol and deliver them to the final brew.

The process of extraction (coffee brew preparation), although fundamentally simple for the consumer (mix then separate hot water

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Table 1
Literature values for cafestol concentration in different brew mechanisms.

	Cafestol (mg/L)	Light roast[a] (mg/L)	Dark roast[b] (mg/L)
Instant	1.9 ± 0.05[c] (150)		
Drip filter	0.12 ± 0.02[c] (150) 3.3[d] (150)		
Boiled	48.3 ± 3.8[c] (150) 13[d] (150)	43.9 ± 1.36 (160)	25.9 ± 3.54 (160)
Turkish	88.7 ± 4.0[c] (60) 17–33[d] (60)	39.1 ± 0.04 (60)	22.8 ± 0.12 (60)
Mocha	37.5 ± 1.3[a] (60) 18 ± 2[d] (60)	26.2 ± 0.60 (60)	19.2 ± 0.37 (60)
French Press	20–27[d] (150) 10–14[e] (70–180)	43.6 ± 0.98 (160)	29.0 ± 0.53 (160)
Espresso	16.7–17.3[c] (60) 40–80[d] (25) 22–30[e] (40–90) 12[f] (50) 26[g] (50)		

All literature values use different cup sizes therefore values are all converted to mg/L to facilitate comparison; cup volume is provided in parenthesis.
[a] I(2) roast color.
[b] I(5) roast color.
[c] Gross et al., 1997.
[d] Urgert et al., 1995.
[e] Buchmann et al., 2010.
[f] Kurzrock & Speer, 2001.
[g] Speer et al., 2000.

and ground roasted coffee) is complicated to predict and requires a number of technical approaches to cover each of the four brew mechanisms tested (Oosterveld, Harmsen, Vorgen, & Schols, 2003; Thaler, 1978; Zanoni, Pagliarini, & Peri, 1992). In this study, cafestol is the compound of interest; cafestol is a lipophilic diterpene that generally resides within the oil phase of coffee and can thermally degrade to form other compounds (Kolling-Speer, Kurt, Thu, & Speer, 1997). The main driving force that needs to be considered when predicting the extraction of cafestol from R&G coffee to the brew is the process of oil emulsification and the removal of physical barriers that would prevent the migration of the emulsified oil (e.g. cell structures or long chain polysaccharide networks) into the brew. It is proposed, therefore, that both the brew mechanism and the physical structure of the coffee (Bell, Wetzel, & Grand, 1996) will impact cafestol brew yield.

The objective of this study was therefore to determine, for the first time, the extraction yield (%) of cafestol from R&G *Coffea arabica* beans at various roasting intensities (roast time) in four brew mechanisms (Scandinavian boiled, Turkish, French press, mocha). This has not previously been documented and will serve to be of value as a reference point for the development of future brew mechanisms and the identification of technical routes to cafestol reduction, and to further explain the complex interaction of brew water and R&G coffee.

2. Materials and methods

2.1. Roast and ground (R&G) coffee

Green coffee beans (*Coffea arabica*) were spread evenly over roasting trays (200 g per tray) and roasted at 190 °C ± 5 °C within a Mono convection oven (Mono, BX, UK). Samples were removed at 10-min intervals to produce a range of products that had been exposed to 190 °C for 0 min, 10 min, 20 min, 30 min, 40 min and 50 min, the resulting roasted coffee beans were designated as raw, I(1), I(2), I(3), I(4) and I(5) respectively to be comparable to light to medium roast intensities in small batch roasting conditions.

Samples were moved to ambient temperature to cool for 2 h then left to degas over 2 days. Roasted coffee beans were stored in folded aluminium bags at 4 °C until required, roasted coffee beans were subsequently ground in a KG 49 grinder (Delonghi, Australia) to a uniform size and sieved (Endecotts, UK) to remove fines and large particulates, R&G coffee was stored at 4 °C until required and samples were analyzed within 5 days of roasting.

2.2. Coffee brew preparation

Turkish coffee was prepared using a traditional Turkish coffee pot (Grunwerg, Sheffield, UK) prepared with 40 g R&G coffee and 300 mL distilled water (Pur1te select, ONDEO, UK). The brew was heated until it had foamed twice, allowed to settle (5 min) then decanted for analysis. Individual cup size was 60 mL.

Scandinavian-type boiled coffee was prepared by adding R&G coffee (40 g) to boiling distilled water (300 mL), allowed to settle (10 min) then decanted for analysis. Individual cup size was 160 mL.

French press coffee was prepared by pouring boiling water (300 mL) on to R&G coffee (40 g) in a glass French press pot (Fisherbrand, US), allowed to stand for 5 min and the plunger was depressed to separate the brew from the grounds. Individual cup size was 160 mL.

Mocha-style brewed coffee was prepared with 40 g R&G coffee and 300 mL distilled water in an aluminium Mocha-maker (Oroley, Spain). Individual cup size was 60 mL.

All coffee brews were prepared at sea level in an air-conditioned room at 21 °C. Brews once prepared were frozen for 24 h at −18 °C and then placed in a Edwards Freeze Dryer Super Modulyo Pirani 1001 (Edwards, Crawley, UK) at −40 °C for 72 h or until a constant weight was achieved (Fisk, Gkatzionis, Lad, Dodd, & Gray, 2009).

2.3. Color

The color of the R&G coffee was measured, as per (Morales & Jiménez-Pérez, 2001) with slight modifications, in the CIE Lab scale (McLaren & Rigg, 1976) (L^*, a^*, b^*) using a tristimulus colorimeter ColourQuest XE (HunterLab, US) after equilibration and calibration (8° standard angle). L^* denotes black to white component, luminosity, a^* denotes + red-to-green component, b^* denotes + yellow-to-blue component (Hunter, 1942) (Standard illumination: D65, colorimetric normal observer angle: 10°, ASTM E308 RSIN Mode, LAV, 1.00 Port, UV Nominal). Samples were placed in transparent square containers and reported as the mean of five determinations at 21 °C.

2.4. Tap density and bulk density

Tap density and bulk density were measured by the ratio of sample weight to tap volume and bulk volume respectively. R&G coffee was poured into a 20-mL cylinder and tapped three times. The volume and weight were measured before and after tapping of the cylinder on the table three times. Bulk density and tap density were then calculated.

The physical structure of the R&G coffee was affected by varying roast intensities. There was no change in the tap density (after compaction), but there was a significant change in the bulk density (measured after free flow with no shaking or settling) (Table 2). Coffee that had been roasted to a L(5) roast intensity was less dense than the coffee roasted to a L(2) roast intensity. Therefore all subsequent experimentations were conducted on a weight basis, to exclude any volume effects on extraction efficiency.

2.5. Cafestol extraction

Two milliliters of 2.5 M KOH (AnalaR, BDH Laboratory Supplies, UK) in 96% ethanol (Fisher Scientific, UK) was added to R&G coffee (200 mg) or freeze-dried coffee brews (200 mg) and saponified at 80 °C for 1 h (GC 8000 series, FISONS instrument, Germany). After saponification, distilled water (2 mL) was added and the water phase was extracted three times with diethyl ether (4 mL, laboratory regent

Table 2
Color parameters (lightness (L^*), a^*, b^* value) and density (tap density and bulk density) of roast coffee by roast intensity.

Roasting intensity	Tap density (kg/m^3)	Bulk density (kg/m^3)	L^*	a^*	b^*
Raw	$493^{a}\pm0.01$	$415^{a}\pm1.27$	$67.3^{e}\pm1.04$	$0.66^{a}\pm0.06$	$14.5^{e}\pm0.42$
I(1)	$514^{a}\pm0.03$	$404^{a}\pm7.21$	$63.8^{d}\pm0.48$	$8.03^{e}\pm0.14$	$22.1^{f}\pm0.41$
I(2)	$504^{a}\pm0.01$	$374^{b}\pm7.65$	$46.6^{c}\pm0.54$	$7.92^{e}\pm0.17$	$10.8^{d}\pm0.28$
I(3)	$497^{a}\pm0.01$	$354^{b}\pm14.7$	$44.2^{b}\pm0.11$	$7.17^{d}\pm0.15$	$8.82^{c}\pm0.31$
I(4)	$490^{a}\pm0.01$	$349^{bc}\pm18.4$	$41.7^{a}\pm0.43$	$6.26^{b}\pm0.10$	$6.86^{a}\pm0.12$
I(5)	$483^{a}\pm0.02$	$322^{c}\pm0.18$	$42.0^{a}\pm0.31$	$6.59^{c}\pm0.16$	$7.63^{b}\pm0.30$

Mean ± standard deviation of values in five replicates. Different letters indicate a difference within a column ($p \leq 0.05$).

Table 3
Cafestol concentration of roast and ground coffee (mg/100 g) and coffee brews (mg/L) by roast intensity and brew mechanism.

Roasting intensity	R&G (mg/100 g)	Boiled (mg/L)	Turkish (mg/L)	French (mg/L)	Mocha (mg/L)
Raw	$642^{a}\pm10.7$	$63.9^{a}\pm1.94$	$45.9^{a}\pm0.04$	$74.4^{a}\pm0.42$	$40.0^{a}\pm1.41$
I(1)	$619^{b}\pm0.9$	$48.2^{b}\pm3.47$	$41.3^{b}\pm0.12$	$53.3^{b}\pm0.68$	$32.7^{b}\pm0.73$
I(2)	$608^{bc}\pm0.32$	$43.9^{c}\pm1.36$	$39.1^{c}\pm0.04$	$43.6^{c}\pm0.98$	$26.2^{c}\pm0.60$
I(3)	$593^{bc}\pm5.68$	$42.5^{c}\pm1.84$	$34.7^{d}\pm0.12$	$25.8^{d}\pm0.14$	$24.1^{d}\pm0.52$
I(4)	$600^{c}\pm12.4$	$35.0^{d}\pm1.64$	$34.4^{d}\pm1.67$	$29.0^{d}\pm6.51$	$22.3^{e}\pm0.27$
I(5)	$595^{c}\pm5.56$	$25.9^{e}\pm3.54$	$22.8^{e}\pm0.12$	$29.0^{d}\pm0.53$	$19.2^{f}\pm0.37$

Mean ± standard deviation of values in five replicates. Different letters indicate a difference within a column ($p \leq 0.05$), R&G is roasted and ground coffee.

grade, Fisher Scientific, UK). Samples were shaken for 10 min at 250 oscillations/min (Denley Spiramix, Thermo Electron Corporation, US) and centrifuged for 5 min at 3000 RPM (CR3i Multifunction, JOUAN, US). Organic phases were pooled then evaporated (15 min, 70 °C, HC502, Bibby Scientific, UK), and residues were dissolved with methanol (HPLC grade, Fisher Scientific, UK) to 25 mL and stored at −40 °C in brown glass bottles with Teflon lids.

2.6. Cafestol quantification

Cafestol extracts were analyzed by HPLC-UV composed of an automatic injector (AS-2055 Plus intelligent sampler, JASCO, Japan), solvent pump (PU-980 intelligent HPLC pump, JASCO, Japan), variable-wavelength UV detector (RI-2031 Plus intelligent RI Detector, JASCO, Japan) and a C_{18} reverse-phase column (250 mm × 4.6 mm, 5 μm). The mobile phase (85:15) was methanol (HPLC grade, Fisher Scientific, UK) and water with an isocratic flow rate of 0.7 mL/min and a detection wavelength of 230 nm (Benassi et al., 2010). The mobile phase was prepared and degassed for 30 min in an ultrasonic bath (F5300b, Decon, UK). Cafestol was quantified by retention time and peak area of authentic standards (ChromaDex, Irvine, USA) using a six-point calibration curve. All samples were within the calibration curve range and repeatability was acceptable at $R^2 > 0.99$. All results are presented on a wet weight basis (mg/L) or (mg/cup).

All samples were prepared in triplicate and analyzed in duplicate. Statistical differences were evaluated by ANOVA-LSD post hoc test (XLSTAT 2011, addinsoft, UK) at a significance level of $p \leq 0.05$.

3. Results

Coffee brews were prepared by four brewing mechanisms to investigate the extraction efficiency of cafestol in each process, and the absolute concentration of cafestol within a brew is detailed in Table 3 on a mg/L basis for each brew mechanism; this is then further detailed in Table 4 on a mg/cup basis, to illustrate parity and to enable comparisons with previous literature. The extraction yield of cafestol from R&G coffee is subsequently shown in Fig. 1 for each roast color and brew preparation.

3.1. Impact of brew mechanism and roast time on cafestol brew concentration

The concentration of cafestol within the R&G coffee significantly reduced with higher roast intensities; this is detailed in Table 3. There was a significant reduction from raw green beans to the lightest roast intensity, I(1) and further roasting at levels I(4) and I(5) gave further reductions in the concentration of cafestol.

The concentration of cafestol in the coffee brews was dependent on both the roast color and the brewing method. The cafestol concentration of the brew ranged from 19.2 to 74.4 mg/L with the highest brew concentration found in the raw coffee sample for all brew preparation methods, further roasting reduced the cafestol brew concentration. French press, boiled and Turkish preparation methods produced the highest cafestol brew concentration and the lowest concentration was found in the Mocha preparation method at all roast colors.

The relative differences in cafestol concentrations were further highlighted on a cup basis (Table 4) as the two highest cafestol brew concentration samples (French and Boiled) also had the highest cup volume. On a mg/cup basis French press and boiled coffee preparations had the highest cafestol level per cup and mocha had the lowest cafestol per single cup serving.

3.2. Impact of brew mechanism and roast time on cafestol extraction yield

When directly comparing the brew extraction yields between different brew preparation mechanisms (French press, Turkish, Mocha, boiled coffee), a marked and significant difference in extraction yield was identified.

Cafestol extraction yield was in the order French > boiled > Turkish > Mocha for both Raw and L(1) coffee, boiled = French > Turkish > Mocha for L(2), and boiled > Turkish > French = Mocha for L(3), boiled = Turkish = French > Mocha for L(4) and for L(5) French > boiled = Turkish > Mocha as calculated by ANOVA-LSD ($p > 0.05$). There was also a strong correlation of roast intensity with cafestol extraction yield (Fig. 1), with green coffee and the lightest roasts having significantly greater cafestol extraction yields than the brews prepared with darker roast coffee (Fig. 1). Of the roasted samples L(1) French press and boiled preparations had the highest cafestol extraction yield (6.5% and 5.84%) and L(5) Mocha and Turkish preparations had the lowest extraction yields (2.42% and 2.88%).

4. Discussion

For all roast intensities, Mocha produced the lowest cafestol concentration; this confirms the work by Gross et al. (1997) who showed that Mocha has the lowest brew concentration when comparing boiled, Turkish and Mocha preparations (Table 1), but is contrary to findings by Urgert et al. (1995) who showed that on a concentration

Table 4
Cafestol concentration (mg/cup) by roast intensity and brew mechanism.

Roasting intensity	Boiled (mg/cup)	Turkish (mg/cup)	French (mg/cup)	Mocha (mg/cup)
Raw	$10.2^{a}\pm0.31$	$2.8^{a}\pm0.00$	$11.9^{a}\pm0.07$	$2.4^{a}\pm0.08$
I(1)	$7.7^{b}\pm0.56$	$2.5^{b}\pm0.01$	$8.5^{b}\pm0.01$	$2.0^{b}\pm0.04$
I(2)	$7.0^{c}\pm0.22$	$2.3^{c}\pm0.00$	$7.0^{c}\pm0.16$	$1.6^{c}\pm0.04$
I(3)	$6.8^{c}\pm0.29$	$2.1^{d}\pm0.01$	$4.1^{d}\pm0.00$	$1.4^{d}\pm0.03$
I(4)	$5.6^{d}\pm0.26$	$2.1^{d}\pm0.10$	$4.6^{d}\pm1.04$	$1.3^{e}\pm0.02$
I(5)	$4.1^{e}\pm0.56$	$1.4^{e}\pm0.01$	$4.6^{d}\pm0.08$	$1.1^{f}\pm0.02$

Mean ± standard deviation of values in five replicates. Different letters indicate a difference within a column ($p \leq 0.05$) on a cup basis, cup size for each preparation: boiled (160 mL), Turkish (60 mL), French (160 mL), mocha (60 mL).

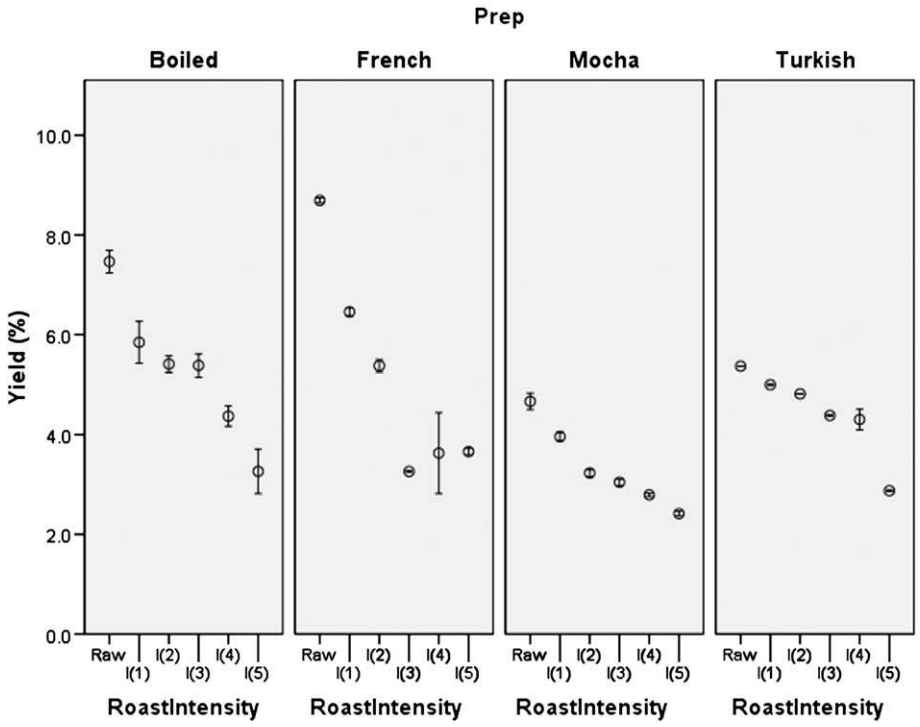

Fig. 1. Cafestol extraction yield by roast intensity and brewing mechanism ±1 standard deviation. Yield = [brew cafestol concentration (mg/L) × total brew volume (L)]/[R&G cafestol concentration (mg/kg) × total R&G (kg)] × 100, where R&G is roasted and ground coffee.

basis, boiled coffee and French press had concentrations of 13 and 10–14 mg/L respectively and that Mocha had an intermediate cafestol brew concentration of 18 ± 2 mg/L when compared to Turkish preparation method (17–33 mg/L). The low concentration of cafestol found in the Mocha preparation is presumed to be due to the fact that the coffee fines and coffee oil (containing the diterpenes) are not significantly transferred to the final brew and are retained in the water tank. The geometry and fill volume will therefore impact transfer rate and may explain Urgert's results.

On both a cup and concentration basis boiled and French press-prepared brews had the highest cafestol concentration, this is due to the elevated levels of physical and thermal stresses imposed on the coffee grounds by these methods and subsequent release of oil and diterpenes into the brew. Turkish style-prepared brews contain an intermediate level of cafestol due to the decanting procedure during preparation, but exceeded that of French press at intermediate roast intensities. Both Urgert et al. (1995) and Gross et al. (1997) showed that French press, boiled and Turkish extraction preparation method can produce high cafestol brew concentrations (boiled, Turkish, mocha and French were studied); Gross et al did not study French press, and found Turkish to be the highest whereas Urgert et al. found French press and Turkish to have the highest concentration. It should be noted that all the data in Table 1 are not truly comparable due to differences in brew geometry, brew volumes and roast color, but do serve to highlight trends that support the general findings shown in Table 3.

There is a small but statistically significant reduction in cafestol in the R&G coffee, with I(5) containing 96% the cafestol of the I(1) coffee, this is presumed to be due to thermal degradation of the cafestol with heating. When considering the coffee brews prepared from I(1) and I(5) roast intensities, the I(5) contains, on average, only 58% of the cafestol that brews prepared from I(1) contain. Given that the original coffee only has a slight reduction in cafestol levels due to thermal damage, there must be a significant impact of roast intensity on the physical release mechanisms occurring during extraction to drive this difference. Kurzrock and Speer (2001) and Urgert et al. (1995) have previously shown only small or no changes in cafestol concentrations with roast intensity, which supports this finding, but do not elude to the impact of roast intensity on the extraction efficiency of cafestol during brewing.

The range of brew extraction yields is shown in Fig. 1, the reason for the significant difference in extraction yield with roast intensity is proposed to be due to changes in the physical structure of the R&G coffee, making it entropically less favorable for the thermal and physical processes to release and emulsify the entrapped oil. As this is driven by the roast intensity, there must therefore be a causal link between heating time and the physical availability of the internal oil reserves of the R&G coffee.

Previously Kurzrock and Speer (2001) and Speer, Hruschka, Kurzrock, and Kolling-Speer (2000) summarized the work by Sehat et al. (1993) and suggested that in a Scandinavian-type brew up to 23% of the total diterpene esters are extracted from the coffee into the beverage, whereas, for espresso and filtered coffee an extraction yield of 0.3% and 2.5% was found.

Sehat et al. (1993) demonstrated that for Scandinavian style brews there was an impact of grind size on extraction yield, with very fine ground coffee having a greater extraction yield when compared to coffee prepared with coarse grind size, which serves to support the conclusion that the physical availability of the cafestol within the R&G coffee has a significant impact on the cafestol extraction yield. Specific numerical comparisons cannot be carried out due to difference in choice of preparation method but the literature results do serve to indicate that the results shown (extraction yield of 2.5%–9.0%) are similar to those previously published (0.3%–23%).

Although this study robustly evaluates the extraction yield of cafestol from within a defined number of samples, it does not address all technologies employed by the coffee industry to create R&G coffee. Future studies should therefore include a more comprehensive investigation into coffee brew extraction kinetics to allow a full understanding of the extraction physics which can then be applied to new brewing technologies (e.g. on demand home brew machines, self-service coffee machines) to control the extraction of cafestol to the brew and minimize consumption by the consumer.

5. Conclusion

Roasting time and choice of brew mechanism impact in-cup delivery of cafestol with French press, boiled coffee and Turkish preparation methods producing higher cafestol concentrations than the mocha preparation method. Higher roasting times led to a 42% reduction in cafestol concentration on a concentration basis within the brews.

The extraction yield of cafestol from R&G coffee is dependent both on the choice of brew mechanism and roasting time, with lighter roast coffee brews having a greater cafestol extraction yield and darker roast coffee brews having a lower cafestol extraction yield.

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